United States Patent Office 3,176,769 Patented Apr. 6, 1965

3,176,769

CONSOLIDATION OF INCOMPETENT EARTH FORMATIONS

Barney R. Treadway, Brea, Joseph F. Chittum, Whittier, and Phillip H. Parker, Jr., San Rafael, Calif., assignors to California Research Corporation, San Francisco, Calif., a corporation of Delaware

No Drawing. Filed Aug. 25, 1964, Ser. No. 392,033
12 Claims. (Cl. 166—33)

This application is a continuation-in-part of application Serial No. 185,533, filed April 6, 1962.

The present invention relates to a method for consolidating incompetent earth formations, such as those adjacent to a well bore, for the purpose of reducing or eliminating invasion of particulate matter into the well bore during fluid production from the formation.

A problem in the production of fluids, such as oil, gas, and water, from an earth formation, is the flow of fine earth particles such as sands, silts, and clays from the surrounding incompetent formation into the well bore with the produced fluids. Particle invasion into a well bore is especially a problem when the formation comprises unconsolidated fine-grained sands and clay or silt particles, such as certain oil-producing sands in California and in the Gulf Coast area. Loose earth particles get into pumping equipment, valves and pipes, causing costly erosion and often making them inoperative. Disposal of produced sand on the surface is also a problem. Further, a successful sand control method permits multiple well completions. In some oil-producing fields, such as the Bay Marchand Field, in the Gulf Coast area, several oil-bearing zones are present. These oil zones are located above each other and are often separated from each other by fluid-impermeable zones. A single well bore may penetrate several of the fluid-producing zones. To produce oil, gas, or water from a single well bore but from several of these zones, a method is required that allows fluid production from all of the zones simultaneously without production of formation particles from any of the zones because equipment in the well bore necessary to produce from all of these zones usually becomes inoperative if formation particles flow into the well bore. It is estimated that several hundred existing wells of one oil-producing company alone could be completed into multiple producing zones if a satisfactory method of sand consolidation were available. A satisfactory sand consolidation method would also increase the number of new wells that could be produced economically because the new wells could be produced from multiple zones instead of from single zones. Involved in multiple zone production is a number of pipes or conduits within the casing, the length of the pipes depending on the zones to be tapped. Unconsolidated sand filling up the casing and the spaces between pipes makes it difficult, and often impossible, to move the pipes in and out of the casing as desired. Accordingly, if the sand can be consolidated within the formation, the sand would not move into the well bore and free movement of the pipes within the casing would remain unimpeded.

A technique for preventing or inhibiting migration of sand into a borehole is known as "plastic sand consolidation." According to this method, there is used a liquid cementing agent, such as an epoxy or phenol-formaldehyde resin, to coat the sand grains, while retaining permeability to fluid flow. The resin, eventually sets up into a chemically unreactive oil-, gas-, and water-insoluble adhesive binding the grains of sand into a porous mass.

A convenient way of providing permeability is to permeate the formation with the liquid resin, and then to flush a fluid through the formation permeated with resin to displace excess resin from the spaces or interstices between particles, while leaving a sufficient amount of resin to bind the particles or grains of sand together. When treating an oil-producing formation, it is often advantageous to remove the film of water from the particles of the formation by means of a suitable agent, such as a surfactant, before coating them with the resin.

It has now been found that plastic sand consolidation of an incompetent formation adjacent to a well bore can be made more effective by the use of a mixture of a unique epoxy resin, hereinafter more fully defined, with a carboxylic acid anhydride, and then copolymerizing in place the carboxylic acid anhydride and the epoxy resin. In accordance with the invention, resin can be distributed throughout the formation to be treated more effectively to give a stronger bond, as measured by compressive strength, between sand particles. Also, it has been found that the present method is particularly adapted to oil-producing formations characterized by the presence of connate or migratory brine or salt water, and which is produced along with the oil. The method of the invention is particularly applicable to oil-producing formations characterized by a temperature above about 150° F.

In carrying out the invention, a mixture of epoxy resin, hereinafter defined, and carboxylic acid anhydride is caused to permeate an oil-producing formation adjacent to a well bore. After the resin-acid anhydride mixture has been placed in the formation, a flushing fluid is injected to displace excess resin mixture and to provide permeability. After the resin mixture is set up into the final hardened form, production of oil is commenced.

In carrying out the invention, certain preferred requirements prevail, as follows:

(1) The epoxy resin component has more than two epoxy groups for each molecule;

(2) More than two of the epoxy groups are of primary nature;

(3) The epoxy-acid anhydride mixture has a viscosity in the range 50–500 centipoises at 150° F.; and

(4) The epoxy-acid anhydride mixture does not prematurely gel. This can be determined by viscosity measurements. For example, a suitable epoxy resin-anhydride mixture maintains a viscosity below about 500 centipoises at 150° F. over a period of 24 hours at this temperature.

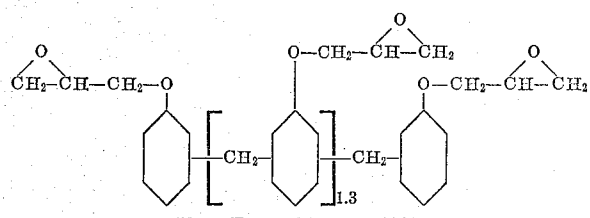
More specifically, the epoxy resin component herein contemplated is substantially oil-insoluble and can be obtained in known fashion such as by condensing epichlorohydrin with a novolac resin of monohydric phenol. Novolac resins containing 2 to 6, and as many as 12, phenolic hydroxyl groups per average molecule have been proposed in the preparation of the type of resin herein contemplated, the use of resins containing a higher number of functional groups being here governed by the viscosity requirements hereinabove specified.

The novolac resins are well-known substances, and many are available commercially. Their preparation is described in the literature, such as in the book Phenoplasts, 1947, page 29 et seq., by T. S. Carswell. In general, these resins are prepared by condensing phenol with an aldehyde in the presence of an acid catalyst. Proportions of phenol and aldehyde in mol ratios of phenol to aldehyde greater than 1.1 and up to 2.5 are taught. As the aldehyde, formaldehyde is preferred, although the use of other aldehydes, such as acetaldehyde, chloral, butyraldehyde and furfural is permissible.

Similarly known is the condensation reaction of the epichlorohydrin with the novolac resin. The reaction is effected at a temperature in the range 140° F. to 300° F. between the novolac resin and at least about 3 mols of epichlorohydrin for each phenolic hydroxyl equivalent of the novolac resin, in the presence of about 1 mol of alkali metal hydroxide per phenolic hydroxyl equivalent of novolac resin. When the reaction is complete, the epoxy

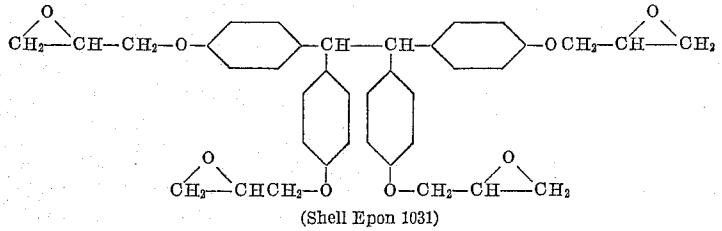
resin is isolated from the reaction mixture by removal of alkali metal salt, of unreacted alkali, epichlorohydrin and water, and purified.

As above indicated, epoxy resins of the type herein contemplated are available commercially. For example, a suitable material available commercially can be represented as follows:



(Dow Epoxy Novolac 438)

As an additional example of a suitable material available commercially is the following:



(Shell Epon 1031)

The preferred curing polybasic acid anhydride is methylbicyclo-(2,2,1)-5-heptene-2,3-dicarboxylic anhydride (known as "Nadic" methyl anhydride). Examples of other suitable anhydrides, aliphatic and aromatic, saturated and unsaturated, are phthalic anhydride, maleic anhydride, hexahydrophthalic anhydride, dichloromaleic anhydride, dodecycl succinic anhydride, pyromellitic anhydrides, adipic anhydride, succinic anhydride, and "Chlorendic" anhydrides (e.g., 1,4,5,6,7,7-hexachlorobicyclo-(2,2,1)-heptene-2,3-dicarboxylic anhydride). The proportions of curing anhydride and epoxy compound are preferably such as to give one anhydride group for each epoxy group, that is, 50 equivalent percent anhydride, determined by adding the total equivalents of epoxy groups and of anhydride groups, expressed in percent. However, the proportions can range from 10 to 70 equivalent percent anhydride.

In the placing of the mixture of epoxy resin and acid anhydride in the formation to be treated, the mixture is caused to permeate the formation by any of the conventional methods. Generally, sufficient mixture is injected into the formation to impregnate it to a radial distance of a few inches to five or more feet from the well bore or in an amount of 2 to 60 gallons of mixture per vertical foot of well hole in the formation to be treated, a generally satisfactory amount being about 1 barrel (42 gallons) per vertical foot.

In a preferred embodiment of the invention, the incompetent formation is pretreated with a water-removing liquid to remove water and thus render it preferentially wettable with the resinous mixture rather than with water.

For this purpose, it is preferred to use an organic solvent which removes water from the formation by miscible displacement. The solvent can then be displaced by the resin or a miscible oil flush before resin injection. Suitable solvents are those which are miscible with brine and either resin or oil. These include low melecular weight ketones, such as acetone and methyl ethyl ketone; aldehydes, such as acetaldehyde, isobutyraldehyde, and formaldehyde; alcohols, such as methanol, ethanol, propanol, isopropanol, isobutanol, and tertiary butyl alcohol; ethers, such as methyl propyl ether, isopropyl ether, and n-butyl ether. The volume of preflush fluid used is generally one to five times the pore volume of the formation to be consolidated, or 3 to 15 barrels per vertical foot of borehole traversing the formation.

Also useful for the removal of water from the formation sands are commonly available surfactants, such as sulfonated naphthenic acids, sulfonated higher alcohols and hydrocarbons, quaternary ammonium salts, silicones, and heavy metal soaps. The surfactant can be mixed directly with the resin mixture of epoxy resin and anhydride, or can be mixed with oil, water, or other fluids and introduced into the formation as an independent pre-conditioning step prior to the injection of the resin mixture. Generally, an amount of surfactant ranging from 0.01 to 1 percent by weight based on resin mixture is satisfactory.

When the formation has been treated with the aforesaid resin to the extent indicated, a resin immiscible flushing fluid is forced through the formation to render it permeable, but yet in an amount to leave the particles with a film of the consolidating resin sufficient to bind the incompetent particles into a porous aggregate mass. Considerable latitude is possible in the injection of flush fluid ranging from immediate application, after the resin is placed, to a delay of several days. The advantage of such flexibility will be readily apparent to those engaged in completing oil wells. The preferred inert flushing fluid is oil, and oil of like character to that to be recovered is eminently suitable. Other examples of inert flushing fluid are water, brine, refined oil, and various organic liquids not compatible with the liquid resin. An amount of flushing fluid which is at least equal to the volume of resin mixture injected, up to ten volumes of the resin mixture, will in most cases be satisfactory, the preferred amounts being 1.5 to 4 volumes of flushing fluid per volume of the original resinous mixture introduced.

The resin under the temperature and pressure conditions prevailing in the well hole will eventually set up into an insoluble, infusible resinous structure through the interaction of the epoxy resin and acid anyhdride. However, in a preferred embodiment, the reaction of the epoxy resin and anhydride is expedited by using in addition to the anhydride, a second curing agent which is an amine. Examples of amine curing agents or catalysts for epoxy resins are benzyldimethylamine, metaxylylene diamine, metaphenylene diamine, diamino diphenyl methane, piperidine, diethylamino-propylamine, diethylenetriamine, dicyandiamine, and methylated derivatives of the above amines. The catalyst is conveniently introduced into the formation following the flushing operation by injecting a solution of the catalyst in oil, such as that used for flushing, the total volume of this latter mixture being again at least equal to the volume of original resin mixture introduced into the formation. Alternatively, the catalyst may be introduced into the formation by a preflush prior to injection of resin, or may in some instances be incorporated with the resin to be injected into the formation. The amount of catalyst used can vary from about 0.1 to 10 percent, preferably around 2 percent, based on epoxy resin and acid anyhdride.

The effectiveness of the method herein contemplated is borne out by the following tests and examples.

For testing there was used a Hassler cell. It comprised a rubber tube, adapted to be fitted with end plugs, provided with screened inflow and outflow openings to prevent loss of sand under pressure, and to permit the flow of fluids through the sand packed in the rubber tube, the plugs being further adapted to be hermetically fastened to a metallic sleeve. The sleeve and rubber tube were of such dimensions as to hold a sand sample five feet long

by one inch in diameter. The sleeve is provided with means for exerting pressure on the sand pack (analogous to the pressure on an incompetent formation from the earth above it, known as over-burden pressure). Associated equipment comprises a pressure tank for storage of fluids prior to their injections, a pump to force the fluid through the sand pack, a bath for heating the Hassler cell, and means, such as a graduate cylinder or flask, to measure the outflow of fluids. After the final injection of the last fluid, the openings of the plugs are closed (shut in) and the treated sand pack allowed to cure.

Example 1

Unconsolidated sand of about 150 mesh was placed in the tube of the Hassler cell. The sand was compacted by vibrating for 15 minutes. Overburden, or sleeve, pressure was held at 3000 p.s.i. and the temperature at 150° F. Diesel oil was flowed through the sand pack to saturate it. At this point permeability was 2.5 darcys.

A cementing agent mixture of 50 equivalent percent Dow Epoxy Novolac 438 and 50 equivalent percent Nadic methyl anhydride is injected in an amount of about 100 ml., i.e., an amount corresponding to one volume of resinous mixture equal to about one-third pore volume of the sand pack. The treated sand is then flushed with No. 5 White Oil [1] in an amount of three to five times the volume of the injected resinous mixture. A pore volume, i.e., 300 ml., of No. 5 White Oil with 2 percent benzyldimethylamine is next injected through the sand pack to initiate polymerization of the consolidating resinous mixture. After about four hours polymerization is complete, and the core is found to have a permeability of 1.2 darcys or about 48% of the initial permeability. The compressive strength of the consolidated sand is about 8000 p.s.i.

Representative samples of the core above, 1½ inches long, were subjected to aging in well bore type fluids for extended periods of time.

One test used to determine durability is the boiling brine test (25,000 p.p.m. of sodium chloride). This test is particularly important. As is known, generally along with the oil there is also produced salt water or brine present in the producing formation as connate or migratory waters. It is therefore highly desirable that the consolidating agent be resistant to the action of brine.

According to the boiling brine test, a number of samples of the core are suspended in boiling brine in a vessel provided with a reflux condenser, the core samples being suspended so as not to touch the bottom of the vessel. The samples are permitted to remain in the boiling brine for varying periods of time. For example, some samples are subjected to the boiling brine for 30 days, while other samples are subjected to the boiling brine for 60 days, and others still for 180 days. The determined values are plotted as a function of time. A value of at least 500 upon extrapolation to three years, a practical minimum, indicates a satisfactory consolidation.

Cores prepared in accordance with Example 1 when subjected to aging in boiling brine for periods up to six months exhibit an average strength of about 2000 p.s.i. This indicates that consolidated formation sand will maintain sufficient strength for periods greater than three years.

In another test, the test cores were immersed in No. 5 White Oil and placed in an oven at 150° F. for 100 days. The cores maintained a strength of about 8000 p.s.i. or 100% of their initial strength.

Further tests were performed to test the durability of the cores by immersing them in a 5 percent hydrochloric acid solution and a 5 percent acetic acid solution, respectively. In both cases, the cores consolidated in accordance with this example exhibited a strength of 4000 p.s.i. for a period of 30 days at a temperature of 150° F.

Example 2

Substantially the same procedure in making the core sample is followed as in Example 1, except that there is employed a mixture by weight of 64.5 percent Dow Epoxy Novolac 438 and 35.5 percent maleic anhydride; the viscosity of this mixture being 88 centipoises at 150° F. After polymerization, the core is found to have a permeability of about 47 percent of the original. The compressive strength is 13,000 p.s.i. After aging in boiling brine for 30 days, the compressive strength is 2000 p.s.i., indicating that formation sand thus consolidated will maintain sufficient strength for a period greater than three years.

Example 3

A core sample is prepared following the procedure of Example 1, except that the cementing agent is a mixture of 50 equivalent percent Nadic methyl anhydride and 50 equivalent percent of an epoxy resin mixture averaging about 2.5 epoxy groups per molecule, and formed from about 16.6 equivalent percent of Shell Epon 1031 and a diprimary epoxide obtained by reacting bisphenol A and epichlorohydrin to a molecular weight of 370. The cementing agent mixture has a viscosity of about 250 centipoises at 150° F. After polymerization, the initial compressive strength is 8500 p.s.i. Permeability is about 52 percent of the original. After aging in boiling brine for 180 days the compressive strength is 2200 p.s.i., indicating that formation sand thus consolidated will maintain sufficient strength for a period greater than three years.

Example 4

As in Example 1, sand was placed in the Hassler cell and vibrated for 15 minutes to compact it. Overburden pressure was maintained at 2500 p.s.i. and the temperature at 200° F. To simulate oil well conditions, brine was first flowed through the sand pack, followed by No. 5 White Oil. Initial permeability was determined to be about 2.6 darcys. There were then injected into the core: 100 ml. of acetone to remove water, 100 ml. of No. 5 White Oil to sweep out the acetone, 100 ml. of the resin mixture of Example 1, 300 ml. of White Oil as flushing fluid, and finally 400 ml. of No. 5 White Oil containing 2% by weight of benzyldimethyl amine. The Hassler cell was then shut in at a sleeve pressure of 3000 p.s.i. and a temperature of 200° F.

At the end of about 65 hours, the core was removed. The core could be lifted from either end, thus indicating complete consolidation due to good resin distribution. The compressive strength of the core was 8,473 p.s.i.

Representative samples of the core, 1½ inches long, were then subjected to the boiling brine test by placing them in boiling brine (25,000 p.p.m. NaCl) for periods of time up to 180 days. After 30 days the compressive strength was 3680 p.s.i.; after 60 days, 1500 p.s.i.; and after 180 days, 1400 p.s.i.

Example 5

Sand similar to that used in Example 4 was placed in the tube of the Hassler cell and compacted by vibrating for 15 minutes. The sand pack was subjected to a sleeve pressure of 2500 p.s.i. and a temperature of 150° F. The sand was saturated with No. 5 White Oil. Initial permeability was about 2.6 darcys. There was then injected into the sand 100 ml. of a resin mixture comprising 52 equivalent percent Nadic methyl anhydride and 48 equivalent percent of epoxy resin derived from bisphenol A and epichlorohydrin, and having an average molecular weight of about 350. Next there was injected 300 ml. of No. 5 White Oil to disperse the resin and establish permeability, followed by 400 ml. of No. 5 White Oil

[1] No. 5 White Oil is a paraffinic hydrocarbon having the following characteristics:

Gravity, ° API	31.5–34.5.
Flash point, Cleveland Cup, ° F.	360 maximum.
Viscosity at 100° F., SSU	106–116.
Pour point, ° F.	+15 maximum.

containing 2% by weight of benzyldimethyl amine. The Hassler cell was then shut in at a sleeve pressure of 3000 p.s.i. and a temperature of 150° F., and allowed to remain shut in overnight. In order to ensure complete cure, the tube containing the treated sand was then placed in an oven at 160° F. and allowed to remain in it for about 65 hours.

The tube was then opened and the core was inspected. The fluid inflow and outflow ends appeared to be soft. Beginning from the inflow end, the first 13-inch portion was poorly consolidated. The next 36-inch portion (13–49 inches from inflow) seemed to be well consolidated. The portion from 49–57 inches was weakly consolidated, while the portion 57–60 inches was characterized by loose sand.

Compressive strength of the 13–49 inch portion of the core was 6,923 p.s.i.

As in Example 4, representative 1½ inch samples of the 13–49 inch portion were subjected to the boiling brine test. After 36 days in boiling brine, the compressive strength dropped to 1280 p.s.i.; and after 60 days, it dropped to zero.

In petroleum oil production, the various fluids, including the resin-carboxylic acid anhydride mixture, may be injected into the formation through a piping stringer or producing tubing placed in the well bore provided with a casing. The formation to be treated may be isolated by positioning a packer just above it, and a packer just below it. Perforations are made in the casing between the packers to provide fluid intercommunication between formation cased well bore and tubing.

In accordance with a preferred embodiment of the invention, the tubing is filled with an inert fluid, such as diesel oil or fluid like that to be produced. The fluid in the tubing serves to establish initial injection, i.e., by its use pumpability of the subsequent fluids into the formation can be determined, and any undesirable fluids in the formation to be treated are displaced.

Treatment then continues by pumping into the tubing the following fluids in sequence, each succeeding fluid pushing the preceding one out into the formation:

(1) A suitable agent, such as acetone, capable of removing water from the sand grains and of rendering them preferentially wettable by the epoxy resin mixture.

(2) A fluid capable of washing the acetone from the tubing and thus preventing it from diluting the epoxy resin mixture, in a quantity like that of the acetone. This fluid is a hydrocarbon oil, such as diesel or crude petroleum oil, in which the acetone is soluble and epoxy resin mixture is insoluble.

(3) Epoxy resin-carboxylic acid anhydride mixture.

(4) Flushing fluid for dispersion of the resin mixture throughout the formation.

(5) Activator or curing agent solution. The activator solution is forced into the formation by a liquid such as diesel or salt water, which later is permitted to fill the tubing and then to provide a hydrostatic head which is at least equal to the formation pressure and thereby preventing the injected fluid from being forced back into the borehole.

In practice, the various fluids are pumped into the tubing at a fast rate consistent with the equipment used and the objectives desired. Accordingly, the resin-carboxylic acid anhydride mixture is injected at a rate sufficiently controlled to effect uniform dispersion of it throughout the formation. It has been found that a resin mixture rate of injection of about $\frac{1}{10}$ barrel to 5 barrels, preferably ½ barrel to 1 barrel, per hour per linear foot of borehole opposite the formation to be treated is satisfactory. The other fluids can be injected at a rate equal to that of the resin mixture ranging up to 20 times the resin mixture rate, a good operable rate being about 6 times that of the resin.

It is also desirable to keep the various fluids separate in the tubing, and minimize intermixing. It is particularly desirable that the resin mixture be forced into the formation relatively uncontaminated. This can be effected by the use of separation means, such as sweep plugs, that keep the various fluids separated. Therefore, it will often be advantageous to insert sweep plugs into the tubing just before and after the plastic-reactive diluent mixture is pumped into the tubing.

The following example illustrates sand consolidation of an unconsolidated oil-producing formation.

Example 6

Plastic sand consolidation using the epoxy resin-Nadic methyl anhydride mixture of Example 1 was applied to a Gulf Coast well over an 11-foot interval at a depth of approximately 7350 feet. The temperature of the formation was about 174° F. It was known that in adjacent wells within a few hundred yards, oil production was not economically feasible from sand corresponding to this interval because the wells sanded up so quickly after they were put on production that the produced oil would not pay the cost of completion and desanding the wells.

The subject well was completed by standard procedure, namely, a well casing was cemented in the well bore with cement pumped into the annular space between the well bore and the casing so that the cement extended above and below the 11-foot interval. The cement was then allowed to set to assure isolation of the subject interval from fluid communication between it and the adjacent zones. The casing was then perforated with 5 holes per foot over the 11-foot interval and a Baker "Model B" packer set in the casing on wire line above the top of the perforated interval. This particular packer permits a tubing string to pass freely through it and seals the tubing string to prevent communication between that portion of the casing opposite the subject interval from the annular space above the packer between the tubing and casing.

The volume of tubing between the surface and formation interval was calculated to be 37 barrels. 47 barrels of diesel were then pumped down into the tubing from a supply tank through the surface piping connected to the tubing. This assured that both the tubing and the casing opposite the subject interval was filled with diesel. A small amount of this diesel was also pumped into the formation to determine the breakdown pressure, that is, the pressure at which the diesel must be pumped to push it out through the casing perforations. It also established the pumping rate that could be obtained with diesel.

33 barrels of acetone were then pumped into the tubing so that 33 barrels of diesel were displaced from the tubing into the formation. This left 33 barrels of acetone in the top of the tubing and 4 barrels of diesel at the bottom. As the next step of the process, another 22 barrels of diesel were pumped at the surface. This pushed the last 4 barrels of diesel and 18 barrels of acetone at the bottom of the tubing into formation. This left 22 barrels of diesel at the top of the tubing and 15 barrels of acetone at the bottom.

A rubber sweep plug was then installed in the tubing just before 11 barrels of resin were pumped in. This addition of resin, of course, resulted in displacing 11 additional barrels of acetone into the formation, leaving in the tubing from the bottom up, 4 barrels of acetone, 22 barrels of diesel, a first sweep plug and 11 barrels of resin. Another sweep plug was then installed in the tubing to isolate the resin. Another 26 barrels of diesel were pumped into the tubing to displace the last 4 barrels of the 33 barrels of acetone into the formation. All of the following 22 barrels of diesel were also displaced into the formation behind the acetone. At this point the tubing contained from the bottom up, 11 barrels of resin and 26 barrels of diesel oil.

Up to this point the rate of injection of all of the fluids had been maintained at about ½ barrel per minute. However, at the beginning of injection of resin into the formation, the pumping rate was decreased to ½ barrel per hour per foot of formation. This made the actual

pumping rate 5½ barrels per hour. 7 additional barrels of diesel were pumped into the tubing at this low rate. At this point there remained 4 barrels of resin at the bottom of the tubing and 33 barrels of diesel immediately above. Next, 31 barrels of activator solution (2% benzyldimethyl amine in diesel oil) were pumped into the tubing. The first 4 barrels of the activator solution were pumped at the resin injection rate of 5½ barrels per hour. However, when all of the resin had been displaced in the formation, the pumping rate of the after flush diesel was increased to 11 barrels per hour or 1 barrel per foot per hour. This rate was maintained until all of the after flush diesel and the activator were injected. In the present example, a sweep plug was installed in the tubing when it was filled with the 31 barrels of activator solution so that all of the activator solution could be pumped out of the tubing by salt water. In this case 33 barrels of salt water were pumped at a rate of 11 barrels per hour to displace the remaining diesel and all but 4 barrels of activator into the formation. These 4 barrels were left in the tubing to prevent contact between salt water and the treated formation during cure of the resin.

Following injection of the resin, diesel, and activator solution, operations were suspended for 4 hours to permit the resin to polymerize into a hard plastic matrix with the desired permeability passages formed therein.

Following withdrawal of the treatment tubing, this well was placed in production. It has produced at the depth-allowable rate authorized by State regulations. That rate is 120 barrels of oil per day and 130 barrels of brine per day.

The following table indicates the material balance of fluids pumped in accordance with this example.

	Contents Tubing Total=37 bbls.		Formation Content
1	Diesel	37 bbls. (full).	Diesel 1-5 bbls.
2	Acetone Diesel	33 bbls. 4 bbls.	Diesel 33 bbls.
3	1st Sweep Plug: Diesel Acetone	 22 bbls. 15 bbls.	Acetone 18 bbls. Diesel 37 bbls.
4	Sweep Plug: Resin Diesel Acetone	 11 bbls. 22 bbls. 4 bbls.	Acetone 29 bbls. Diesel 37 bbls.
5	Diesel Sweep Plug: Resin Sweep Plug: Diesel Acetone	1 bbl. 11 bbls. 22 bbls. 3 bbls.	Acetone 30 bbls. Diesel 37 bbls.
6	Diesel Sweep Plug: Resin Sweep Plug.	1 bbl. 25 bbls. 11 bbls.	Diesel 22 bbls. Acetone 33 bbls. Diesel 37 bbls.
	Decrease pump rate to ½ bbl./hr./ft. (5½ bbls./hr.)		
7	Diesel Resin	7 bbls. 1 bbl. 25 bbls. 4 bbls.	Resin 7 bbls. Diesel 22 bbls. Acetone 33 bbls. Diesel 37 bbls.
	Increase pump rate to 1 bbl./hr./ft. (11 bbls./hr.)		
8	Sweep Plug: Activator Diesel	 31 bbls. 6 bbls.	Diesel 27 bbls. Resin 11 bbls. Diesel 22 bbls. Acetone 33 bbls. Diesel 37 bbls.
9	Salt Water Sweep Plug: Activator	33 bbls. 4 bbls.	Activator 27 bbls. Diesel 27 bbls. Resin 11 bbls. Diesel 22 bbls. Acetone 33 bbls. Diesel 37 bbls.

We claim:

1. Process for the cementing together, in a permeable aggregate, of loose earth particles contained in an incompetent formation adjacent to a borehole for the recovery of fluid, which comprises pre-flushing the formation with a water-removing liquid to render the formation preferentially wettable by the cementing agent, impregnating the flushed formation with a liquid resinous mixture of an epoxy resin and a polycarboxylic acid anhydride copolymerizable therewith to the infusible insoluble state, flushing the impregnated formation with an inert liquid to provide permeability to the formation and leave a film of the resinous mixture around the earth particles, then contacting said film with an epoxy resin amine curing agent, said epoxy resin having more than two epoxy groups in primary position per molecule, and said resinous mixture having a viscosity in the range 50 to 500 centipoises at 150° F.

2. Process according to claim 1, wherein preflushing of the formation is effected with acetone.

3. Process according to claim 1, wherein the polycarboxylic acid anhydride is present in proportions ranging from 10 to 70 equivalent percent.

4. Process according to claim 1, wherein the amine curing agent is used in an amount of 0.1 to 10 percent by weight based on epoxy resin and polycarboxylic acid anhydride.

5. Process according to claim 4, wherein the amine curing agent is benzyldimethylamine.

6. Process according to claim 1, wherein the amount of preflushing fluid used ranges from 1 to 5 times the pore volume of the formation.

7. Process according to claim 6, which includes after impregnation of the formation with resinous mixture flushing, the impregnated mixture with an inert flushing fluid which is equal to the volume of resinous mixture up to 10 volumes thereof.

8. Method of consolidating an oil-producing earth formation of loose sand particles characterized by a temperature above about 150° F. and traversed by a borehole provided with producing tubing having fluid intercommunication with said formation, which comprises filling said tubing with an inert hydrocarbon fluid to establish initial injection, and then effecting the following operations in sequence:

(*a*) pumping into the tubing and into the formation a water-removing liquid to render the formation preferentially wettable by epoxy resin;

(*b*) pumping into the tubing and into the formation an epoxy-resin immiscible hydrocarbon fluid to wash the water-removing liquid from the tubing;

(*c*) pumping into said tubing and into the formation a liquid resinous mixture of an epoxy resin and a polycarboxylic acid anhydride copolymerizable therewith, said epoxy resin having more than two epoxy groups in primary position, and said resinous mixture having a viscosity of in the range 50 to 500 centipoises at 150° F.;

(*d*) pumping into the tubing and into the formation a flushing fluid immiscible with the epoxy resin to spread the aforesaid liquid resinous mixture through the formation and thus provide permeability;

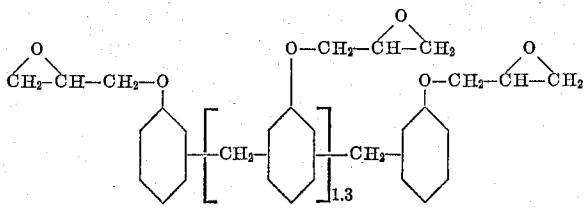
(*e*) pumping into said tubing and into the formation a solution of an epoxide resin curing agent; and

(*f*) maintaining within said tubing a hydrostatic head at least equal to the fluid pressure in the treated formation.

9. Process according to claim 8, wherein the water-removing agent is acetone.

10. Process according to claim 8, wherein the curing agent is an amine.

11. Process according to claim 8, wherein the epoxy resin is of a type represented by the formula:



the polycarboxylic acid anhydride is methylbicyclo-(2,2,1)-5-heptene-2,3-dicarboxylic anhydride; the curing agent is benzyldimethylamine.

12. Process according to claim 11, which includes forcing the liquid resinous mixture into the formation at a rate of ½ barrel to 1 barrel per hour per vertical foot of borehole traversing the formation.

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CHARLES E. O'CONNELL, *Primary Examiner.*